

ANL/HFEF-007

THE  
SODIUM PROCESS DEMONSTRATION  
- A STATUS REPORT

DOE

by

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MARTIN F. HUEBNER

HOT FUEL EXAMINATION FACILITY  
REACTOR EXPERIMENTS AND EXAMINATION DIVISION  
ARGONNE NATIONAL LABORATORY  
IDAHO FALLS, IDAHO

(Partial Draft Issue)

November 1983



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\* Text of this section to be furnished by others outside of the HFEF organization.

\*\* Page designations for sections of this draft version. "SDD" refers to "System Description-Draft," "ATD" refers to "Acceptance Testing-Draft," etc.



## ABSTRACT

The Sodium Process Demonstration (SPD) Project facility is a chemical-process pilot plant designed and operated at Argonne National Laboratory-West (ANL-W) to remove sodium metal residues from metallic materials and equipment.

A combination melt/evaporation process is used to separate the sodium from sodium-bearing items. The sodium is then oxidized to sodium monoxide via a oxidation-calcination process. The monoxide can be stored for disposal or converted to sodium carbonate in another facility.<sup>(15)</sup>

The SPD is currently used to process nonradioactive items. It was designed so that when it is installed in a suitably shielded process facility, it will process sodium-containing radioactive equipment and materials from the nation's Liquid Metal Fast Breeder Reactor program. Thus used, the materials and equipment that are freed of sodium, and the sodium oxide, can be disposed of separately via conventional waste-disposal techniques.

In 1983, the operators of the SPD facility completed a MEDEC\* Process Test program. This confirmed the SPD's ability to remove sodium films, to remove sodium from crevices and holes, to process equipment items containing bulk sodium, and to convert sodium to the monoxide via the calcination process.

Modification of the SPD to a low-level radioactive-sodium waste facility at ANL-W is planned in the future.

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\* Melt/Evaporate/Drain and Calcine.



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These persons were all instrumental in conceiving, exploring, and evaluating concepts which led to establishing the SPD Project, and orchestrating the funding and design and the many other tasks which led to successful construction of the facility. Throughout the MEDEC Test program, Mr. Larsen was a resource of technical expertise often consulted by SPD Operations long after his official role as SPD Project Manager was over. He was always very generous in sharing his knowledge, personal time, and in giving sagacious counsel.

The author also is grateful for the support of HFEF personnel during the acceptance/operations phase, especially J. P. Bacca, HFEF Complex Manager; F. DiLorenzo, Operations Manager; Bob Phipps, Experimental Coordination and Control Manager; and M. D. Carnes, who served as SPD Qualification Engineer during acceptance testing. These men gave the SPD operations staff tangible support and useful guidance throughout the acceptance testing and the prosecution of the MEDEC Test program.

Finally, and especially, the author is deeply grateful to the following, for without their technical expertise, unwavering professionalism, and their on-the-job vast commitment of personal effort, the MEDEC Test program might not have been as successful:

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R. Carlson, Electrical Engineer, ANL-W Site Engineering,\* who was assigned to oversee the process control responsibilities, and to the absolutely prodigious job of doing all the process control computer



programming for the SPD project. Not only did his competence pay off in dispelling concerns about the practicality of computer-controlled chemical processing, but without his tremendous personal commitment for over 18 months, none of the project and test program milestones would have been achieved as scheduled.

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\* Now associated with Specialty Electrics Inc. Hailey, Idaho.





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## SUMMARY DESCRIPTION

Most of the process equipment for the Sodium Process Demonstration (SPD) is contained in Bldg. 789 of the ANL-W site. The building is a 32-ft x 75 ft structure on the east side of the site. Adjacent to this building are two auxiliary buildings. One is a temporarily installed trailer with office space for the SPD Operations staff, sanitary facilities, and blueprint files. The other small building (789A) houses an emergency electrical generator and some auxiliary process cooling equipment.

The process has been given the acronym MEDEC for melt/evaporate/drain and calcine. The process equipment consists of five major items, all installed in an enclosed process cell that is accessible for preparatory operations and maintenance. (See Fig. 1-pod.)

The five major equipment items are: (1) A cylindrical melt/evaporate (M/E) vessel 54 in. ID and 8 ft in. inside usable height, in which to melt or evaporate metallic sodium. The M/E vessel is connected by duct to (2) an air-cooled condenser for condensation of sodium vapor. Piped to the bottom of the condenser is (3) a small drain tank to collect sodium condensate. The other equipment items are (4) a 350-gal cylindrical tank for sodium storage and calciner feed, and (5) the calciner, a horizontal rotating drum for sodium oxidation. These five major items are interconnected with sodium process lines, inert-gas pressurization lines, air lines, and miscellaneous electrical, control, and instrumentation services. Outside the building are located some cryogenic inert-gas dewars.

The entire process is controlled by a small computer whose actions are specified and monitored by a qualified SPD process operator. The computer is programmed to prompt the SPD operator to further actions when certain decision points in the program are reached.



## ACCEPTANCE TESTING

Within the ANL site, the organization assigned to operate the SPD Facility is the Hot Fuel Examination Facility (HFEF). However, construction and initial acceptance testing were assigned to ANL-W Site Engineering. In early 1982, HFEF began assisting in acceptance testing at a stage where the MEDEC process components had been installed and checked out as individual components, but the control computer was not yet installed and all integrated system checkouts remained to be performed.

At this stage, HFEF assigned one qualification engineer, one electrical engineer, two hot-cell technicians, and an electronics technician to work under the SPD Project Manager of ANL-W Site Engineering. HFEF also appointed two people to the SPD operating staff -- an Operations Supervisor and a Process Systems Engineer. These two were assigned to do intermittent surveillance of the acceptance work and to train the acceptance technicians who would eventually become SPD operators.

A qualification plan had been prepared by the SPD Project Manager to define acceptance of SPD components and systems. This plan was adapted as an HFEF Process Work Sheet document, a format with which the HFEF personnel were familiar. Additional qualification tests were written as necessary; and equipment changes were documented by a "Change Request Form - SPD."

The acceptance-testing phase continued until formal HFEF takeover of the facility in mid-July 1982. The most noteworthy events during this phase were the following:

The sealing surfaces between the calciner drum - which rotates - and the mating stationary surfaces were found not to be parallel. (A great deal of effort was eventually required to realign, support, and eventually reface these surfaces for effective sealing.)

The SPD process control computer was installed, and was tested in conjunction with the process instrumentation and with the electrical strip heaters of the M/E vessel. Much of the software for process control remained under development.





The gaseous fire-suppression system in the process-equipment pit was tested with nitrogen. The test showed that the oxygen concentration in the pit would only decrease to about 13% over a 10-min interval. This was not considered adequate for sodium-fire suppression, so the fire-suppression system was converted to argon. (Subsequently, the inlet ducting for the condenser blower was extended to the bottom of the process-equipment pit to ensure that no argon gas concentration could build up in the pit in the event of an undetected argon leak.)

To evaluate the temperature distribution inside the M/E vessel during its acceptance testing, a temperature-measuring fixture was designed and installed. The test results corroborated earlier speculation that the bottom of the vessel would be significantly cooler than the rest. Design work to add supplemental heating was initiated.

Preparations for initial sodium operations included writing of step-by-step procedures including those for emergencies; installation of protective equipment, including a sodium first-aid burn kit, a Scott Air Pak rescue breathing apparatus, fire extinguishers, and backup portable oxygen analyzers; and drafting of the Fire Department preplan for the SPD.

At the end of the acceptance-testing phase, uncompleted modifications were documented in a formal "Change Notice" listing.

In early 1982, during the latter portion of the construction/acceptance phase, ANL-W Site Management, including the Manager of the Sodium Waste Technology Program, reviewed the progress being made on the SPD Facility/MEDEC Process Systems. Site Management concluded that to meet the goal-dates for completing acceptance testing and the commencement of operations, the eventual operators of the facility, the Hot Fuel Examination Facility (HFEF) organization, would have to immediately become involved to assist in acceptance testing. An additional and significant concern expressed at that time was also that the computer programs required to support operations were not available and would not be available for a long time.

One engineer from the HFEF Operations Support Group, who is experienced in mechanical equipment design and field installation check out,



was assigned to the SPD project as the Qualification Engineer. Two HFEF Hot Cell Technicians, an HFEF Electrical Engineer, and an Electronic Technician from the EBR-II Critical Systems Maintenance Group (now the HFEF I&C Group) were also immediately assigned full time to the SPD Project. These men reported to and worked directly for the SPD Project Manager.

In addition, the SPD Operations Supervisor and Process Systems Engineer (experienced HFEF Operation staff engineers who were assigned to ultimately oversee SPD operations) began intermittent surveillance activities of on-going construction as reported in Ref. 2.

With the HFEF personnel involved, the pace accelerated in equipment check out and acceptance work. The overall qualification plan (1) used by SPD Project Management to define acceptance of systems and individual components, was also adapted as an HFEF Process Work Sheet document. This is a format with which the newly-assigned HFEF personnel were familiar.

In addition to their surveillance function, the HFEF Operations Engineers assigned to SPD began a bootstrap "Learn-While-You-Turn" training program for the mechanical technicians assigned to the Project. Some of these technicians so trained would ultimately become the SPD facility operators.

Qualification tests supporting the acceptance qualification plan were written, approved, and performed. Changes found necessary were documented per a "Change Request Form-SPD." This work lead shortly to the acceptance of the condenser cooling system, acceptance of the mechanical rotation equipment for the calciner, and also for process piping and valving. As mentioned previously, the sealing surfaces between the calciner drum - which rotates - and the mating stationary surfaces were found not to be parallel, and a great deal of effort was required to realign, support, and eventually reface these surfaces for effective sealing.

The SPD process control computer was installed and checkouts commenced between it and the process system's thermocouples, and flow and



pressure transducers. A preliminary test was run on the electrical strip heaters of the melt/evaporation M/E vessel. The emergency electrical generator was checked out, and a temporary trailer (to provide an SPD Operations office) was installed just north of the SPD facility. The free-discharge flow test of the nitrogen supply cryogenic dewars was completed.

A test was subsequently performed of the gaseous fire-suppression system for the process system equipment pit using nitrogen gas. The test indicated oxygen concentration would decrease in the pit only to about 13% over a 10-minute interval. This was not considered to be adequate for potential sodium fire suppression; therefore, plans were made to convert the fire suppression gas system to argon.

To evaluate the temperature distribution inside the M/E vessel during M/E vessel acceptance testing, and related heatup/operation tests, a temperature-measuring fixture was designed and installed. Test results subsequently corroborated earlier speculation that the bottom of the vessel would be significantly cooler than the rest of the vessel. Three kW of electrical power was subsequently installed on the bottom exterior of the vessel and the temperature distribution test was repeated. This augmented heater power helped to raise the temperature of the vessel's bottom at the work piece location to be somewhat closer to that of the rest of the vessel, but did not completely correct the situation.

Acceptance work continued, with efforts concentrating on grooming the process control system, completing the installation of thermal insulation, and modifying the pit's fire-suppression system to use argon gas.

With a proposed turn-over date to HFEF Operations by July 31, 1982 (to support initial sodium operations by that date) a formal Facility Readiness Review was held in mid-July to discuss the following:

- Facility Equipment Qualification Status,
- Facility Safety Plans, and the Facility Preliminary Safety Analysis Report,
- The status of the computer programs necessary to operate the M/E portion of the MEDEC process system,



- The status of the required operating and maintenance procedures, and
- The availability of experimental sodium-filled test specimens and test procedures.

The conclusion was reached that the facility was ready for operation as justified by the following reports:

- All procedures\* required for the initial sodium operations<sup>(3)</sup> were on hand and ready for approval. These included procedures for prestartup inspection and closure of the process cell, loading (and unloading) test specimens in the M/E vessel, removing and replacing the M/E vessel's lid, checking the integrity of the vessel's lid seal, plant warmup, sodium melting, sodium evaporation, and system cooldown/shutdown. Processing operations would be conducted via these procedures by combined computer/ manual operations at the computer console. (This computer/manual mode of operation persisted until very late in the MEDEC Test program when software development, based on operating experience, supported computer control of the entire process.)

- A comprehensive procedure\* covering system response to process alarms and emergencies, including operator backup response, was also developed and approved. The HFEF staff assigned to SPD Operations had reviewed these procedures, and the crew was suitably familiar with the procedures.

- A test procedure\* had been written to support processing the first experimental sodium-bearing test specimen,<sup>(4)</sup> and the operators were familiar with the procedure.

- The test specimen (a small stainless beaker containing several hundred grams of sodium) had been prepared in the Lab and Office (L&O) inert gas filled glove box. The transfer container (with inert gas atmosphere, for transferring the specimen from the L&O and receiving it in SPD) was on hand.

- The SPD Preliminary Safety Analysis Report<sup>(5)</sup> was revised by SPD Operations. It was transmitted to ANL-W officials of the Sodium Waste Technology program, who in turn relayed the document to DOE for information.

\* The format used for these operating and test procedures was HFEF's Process Work Sheet format, administered under the HFEF Management Plan (12)





- An ANL-W Safe Work Permit covering the handling of the test specimen's related argon and sodium hazards had been approved by the ANL-W Safety Engineer. The protective equipment required for this work was on hand, as was a sodium first aid burn kit, a Scott Air Pak rescue breathing apparatus, the requisite fire extinguishers, and backup portable oxygen analyzers.

- The inlet ducting to the condenser blower was extended to the bottom floor level of the process equipment pit. This ensured that no argon gas concentration hazardous to personnel could build up in the depressed floor area of the process equipment pit in the event of an undetected argon gas leak.

- A minor defect in the M/E vessel (caused by an unsuccessful stud welding effort to install some thermocouple guards) had been repaired per qualified Quality Assurance procedures, and inspected by ANL-W Quality Assurance personnel.

- The pit fire suppression system for the process equipment pit had been converted to argon gas. Although a larger capacity pressure regulator had yet to be installed, the present regulator was adequate for use.

- The two process-system oxygen analyzer units were cross-connected. This permitted evaluating the effectiveness of nitrogen gas purge operations from the start to finish of M/E process operations.

The M/E vessel had passed a stringent vacuum leak test, and a dry run of the procedures for nitrogen purging had demonstrated their effectiveness.

- The Fire Department Preplan for the SPD had been drafted and was considered adequate.

- Although there were a great many unresolved or uncompleted items, the procedures, the recent acceptance-test results, and plant modifications made (to that date) to the SPD facility supported commencement of facility sodium test operations.

HFEF assumed operational responsibility for the SPD facility during mid-July 1982. The items that had been recommended to SPD Project Management for modifications yet uncompleted were documented in a formal "Change Notice" listing (see "Improvements"). The acceptance testing phase of the SDP Project was therefore announced "Complete."



## SUMMARY OF OPERATION

The MEDEC (Melt/Evaporation/Drain and Calciner) process equipment complex was constructed in the Sodium Process Demonstration (SPD) facility at Argonne National Laboratory-West (ANL-W) and is contained in a single 32-ft x 75-ft building (Bldg. 789).

Adjacent to it are two smaller buildings. One is a temporarily installed trailer with office space for the SPD Operations staff, sanitary facilities, and blueprint files. The other small building (Bldg. 789A) houses an emergency electrical generator serving the facility and some auxiliary process cooling equipment.

The process equipment consists of five major items (see Fig. 1 - pod) installed in a process cell that is enclosed but accessible to personnel. Three of these items are a cylindrical melt/evaporator (M/E) vessel about 54 in. across (ID) and 10 ft high (with about 8 ft usable inside height) in which to melt/evaporate metallic sodium, a duct-connected air-cooled condenser for sodium vapor condensation, and a small sodium drain tank piped to the bottom of the condenser to collect sodium condensate. The balance of the system is composed of the sodium storage/calciner feed tank (a cylindrical tank of about 350-gal capacity) and a horizontal rotating-drum sodium-oxidizer machine called a calciner. These process units are interconnected with sodium-process lines, inert gas pressurization lines, air lines, and miscellaneous electrical, control, and instrumentation services. Some cryogenic inert-gas dewars are located outside of the SPD Building.

The entire process is operated by, and under the direct control of, a small process-control computer whose actions are specified, monitored, and overseen by a qualified SPD process operator. The computer programming is such that the computer will "prompt" the SPD operator to specify further actions when certain decision points in the program are reached.

As depicted in Fig. 2-pod, the possibilities for various kinds of process operations are complex. These can be divided into two main subdivisions; these are activities pertaining to (1) Sodium melt/ evaporation/drainage and liquid-sodium transfers and (2) Calciner operations.



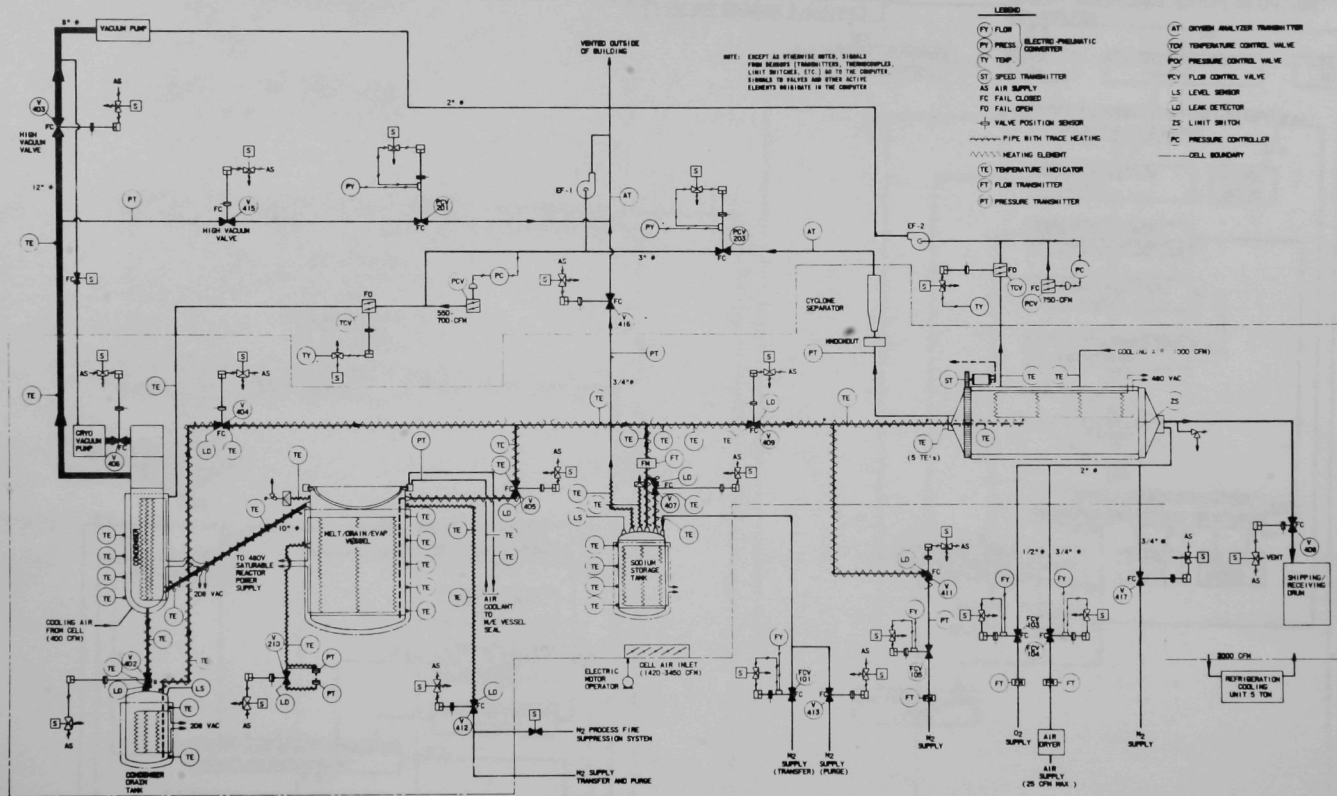
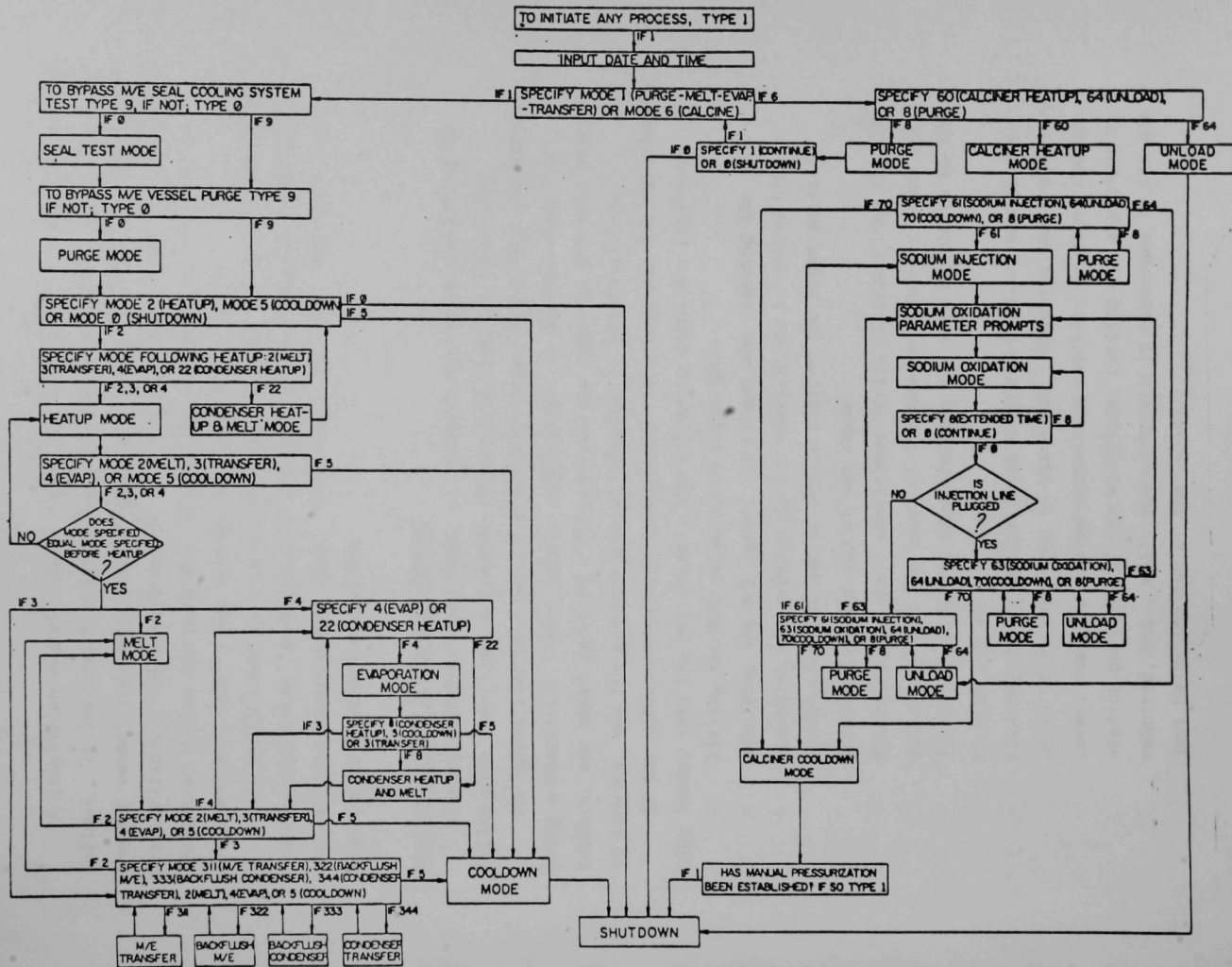


Figure 1-pod - SPD Process System









A simplified description of the first subdivision of these operations follows. It includes loading the sodium-containing workpiece(s) into the M/E vessel, process-system startup and heatup to 400°F, melt/drain/and pressurized-gas-initated transfer of the sodium to storage, the subsequent additional heatup and pump-down evacuation of the M/E vessel, sodium evaporation at 900°F, and system cooldown and shutdown.

This is followed by a description of calciner operations, including startup, calcining (oxidizing) of sodium, calciner product unloading, and calciner shutdown/cooldown. This section will not discuss all the alternative processing options.

#### Startup of Melt/Evaporate System

During shutdown conditions, all process equipment and piping is filled with inert gas to prevent undesirable chemical reactions.

For plant startup, various electrical power panels are energized using a checklist. These panels supply power to motors, electrical heaters, and other services. Pressurized-gas supplies are then placed in service, and the process-control computer is placed on line.

If the sodium-containing workpiece or test specimen has not already been loaded into the M/E vessel, the following steps are required:

- Filling the M/E vessel with argon gas.
- Unbolting the M/E vessel lid clamps and removing the lid.
- Operating the process cell's ventilation to ensure proper ventilation but not to unduly disturb the argon gas/air interface at the top of the vessel.
- Maintaining a small continuous inflow of argon into the M/E vessel so that the level of the heavier-than-air argon gas fills the vessel to approximately the same elevation as the flanged top of the vessel.
- Lowering the workpiece (with overhead handling equipment) until it is submerged in the argon. Removing the protective covers over any sodium-containing cavities or other sodium-bearing portions of the workpiece, cutting off any pipes as required, etc., while the workpiece is submerged in the gas just below the top of the vessel.



- Lowering the workpiece to its processing set-down position on the grating at the bottom of the cylindrical wall of the vessel. This will expose the workpiece subsequently to peripheral radiant heating from the vessel's wall. The item set-down is oriented to facilitate sodium drainage.
- Replacing and clamping down the M/E vessel lid.

With all preoperations complete, the operator initiates process operations at the computer control console and specifies subsequent activities via an interactive process-control program.

#### Plant Heatup for Sodium Melting

The operator performs some process-system checks such as confirming that the gas inside the vessel is below a certain oxygen limit; then plant heatup is initiated. Power is applied to the vessel electrical strip heaters (and other equipment needing heatup such as sodium lines) in a preplanned heatup sequence. The sequence is controlled by computer direction of a saturable reactor.

At the sodium melting temperature of 400°F, the heatup levels off for a melt interval prespecified by the operator. The interval is timed to last until prior calculation, or experience, indicates that all sodium has melted and drained from the workpiece into the bottom of the M/E vessel.

The operator then specifies the operations for removing the melted sodium from the vessel. The computer lines the process system up for sodium transfers. Valves are opened in sodium lines, vent lines, and inert-gas lines. The M/E vessel is gas-pressurized slightly above the pressure in the storage tank. Melted sodium is gas-pressure-transferred from the M/E vessel through heated process lines into the sodium storage tank.

If there is a concern about sodium-oxide deposition in the line that might cause line blockage, the operator can specify sodium flows in the the reverse direction (line backflushing) to dissolve such incipient blockages.



### Heatup for Sodium Evaporation

After the melted-sodium transfers have been made, the operator can use the computer control console to energize the high-vacuum pump and commence the heatup of the M/E vessel. The computer again directs the electrical saturable reactor to increase power to the M/E heaters in a predetermined program.

The M/E vessel is heated to  $\sim 900^{\circ}\text{F}$ , with heater power decreased near the end of the heatup to prevent overshoot of the temperature setpoint. At  $\sim 900^{\circ}\text{F}$ , the computer commences a preselected time interval for evaporation. At the end of this interval, the operator can opt for more evaporation time if necessary.

During the evaporation phase, the vacuum pump remains operating, with the absolute pressure maintained at  $\sim 10\text{-}50$  microns depending on the evaporation rate from the workpiece. The air-cooled condenser is operated to promote sodium-vapor transport from the M/E vessel and condensation in the condenser.

### Cooldown/Shutdown

At the completion of evaporation, the operator may specify plant cooldown, with the high vacuum maintained. During this period, certain heaters attached to the upper portion of the M/E vessel remain energized to prevent the redeposition of unwanted sodium-vapor inside the vessel.

At  $400^{\circ}\text{F}$ , the vacuum pump is deenergized and the M/E vessel is automatically backfilled with inert gas. The operator can then choose that the condenser be heated to melt sodium solidified in it. During this operation, condenser air cooling is secured and external condenser heaters are energized. As a result, any solidified sodium melts and is collected in the sodium drain tank below.

At  $300^{\circ}\text{F}$ , the control systems can be deenergized and the M/E system shut down using a checklist. Internal M/E system pressure has been controlled automatically during the cooldown and will be manually controlled during the subsequent shutdown.

After plant cooldown to  $\sim 120^{\circ}\text{F}$ , the M/E vessel lid can be unbolted and the sodium-free workpiece can be removed.



## Calcination

During shutdown conditions, the calciner pressure is kept slightly above atmospheric by means of a small continuous purge flow with dry inert gas. This protects the hygroscopic sodium monoxide from air humidity, which would cause sodium hydroxide formation in the bed of monoxide granules in the calciner drum.

Calciner startup requires supplying electric power to the heaters on the sodium storage tank and interconnecting lines, and energizing power panels and other services, per a checklist. The process control computer is then put on line to start calciner rotation and external heatup of the rotating drum via electric heaters.

When the sodium storage tank, the sodium-process lines, and the calciner are at operating temperatures, the computer requests additional processing information. The operator, via the computer control console, selects such process variables as sodium batch size (in gallons), time of mixing of the sodium in the bed after sodium injection, drum rotation speed, etc., for the mixing operation.

After the sodium has been injected and is well mixed in the sodium monoxide granules in the bed, the operator selects the process variables for the upcoming oxidation-calcination phase. These variables include oxidation reaction time, drum speed, flow rate of oxygen and air mixed in the reaction-gas supply line, and reaction-endpoint oxygen level in the off-gas (effluent) line.

To oxidize the sodium, a prespecified flow in terms of cubic feet of air/minute is injected into the drum through the reaction-gas line. This air mixes with the ongoing nitrogen purge of the sodium line that flows through that line after sodium injection. The air-nitrogen mixture is introduced into the nitrogen-filled atmosphere of the calciner drum. As the further-diluted oxygen contacts the liquid-sodium films on the monoxide granules, the oxidation-calcination reaction commences. The temperature of the drum and its contents increases because of the exothermic oxidation reaction. External drum heating and external cooling-air flows are used to keep drum temperatures within the desired range.





The reaction temperature rises slightly (in the drum) until most of the sodium has reacted, then it decreases towards the original preoxidation value. The temperature decrease corresponds fairly closely to a rise in the oxygen level of the gas effluent leaving the drum through the off-gas line.

When the specified reaction-time period has ended, the operator can opt for several alternatives, the most likely of which is to unload some monoxide.

### Product Unloading

Calcliner efficiency requires about 20% of the entire drum volume to be occupied by sodium monoxide. Too high a bed level can result in some of the bed being insufficiently exposed to the in-drum atmosphere. This could result in poor conversion of sodium to sodium monoxide. Too low a drum volume of the bed can cause excessive bed tumbling action, resulting in excessive reduction in the size of the bed granules. Thus, after conversion of a sodium batch to monoxide, unloading of the drum is required.

When the operator selects the calciner-unloading option, the drum is run in reverse direction for a small, predetermined number of revolutions. A mechanical scoop inside the drum scoops up a portion of the bed in each revolution. This monoxide goes to a hopper outside the drum for product unloading. The product falls into an inert-gas-filled steel drum.

### Cooldown

Cooldown is controlled by the computer after the operator selects this option. After the calciner has cooled, the shutdown purge flow of nitrogen is manually reestablished, and the system is lined up for shutdown using a checklist.



## OPERATIONAL EXPERIENCE

The "Operational Experience" phase of the SPD Project began at the completion of construction and acceptance testing, with the corresponding assumption of operational responsibility by the HFEF organization. This new phase was marked by the start of in-facility tests with sodium, which initiated the MEDEC Test Program at ANL-W.

As originally defined<sup>(6)</sup> the MEDEC Test Program had four primary test objectives:

- To conduct melt/evaporation operations on sodium-bearing test items and calcine the sodium thus collected into storable sodium monoxide.
- To remove the sodium heat-transfer bond from unirradiated EBR-II blanket rods containing depleted uranium.
- To remove the sodium from an EBR-II cold trap.
- To process selected organic materials in the M/E vessel to investigate the deleterious effects on the rest of the M/E system.

This last item was included in the test program because of the expectation that the MEDEC process equipment would (in a few years) be reinstalled in a shielded facility. In this proposed installation,<sup>(6)</sup> the MEDEC system was to receive and process a number of sodium-bearing waste materials now in interim sodium-waste storage. The likelihood of plastic residues in these feed materials to the M/E process was high, hence the need to evaluate the effect of organic materials on the rest of the MEDEC system.

With the suspension of interest in a shielded process facility,<sup>(7)</sup> the need for the last MEDEC test objective listed was eliminated. The "Operational Experience" phase of the MEDEC Test program therefore concentrated on fulfilling the first three objectives listed.

The MEDEC Test Program was conducted as a series of miniature processing campaigns, starting with processing of small-sized sodium test samples in the M/E vessel, and proceeding to larger-sized samples and multisample tests; then investigating the evaporation of sodium from sodium-filled crevices; then proceeding with vapor conductance tests



(where evaporation through ducts connecting a pool of sodium to the interior environment of the M/E vessel was studied), bulk-sodium operations, calcination tests, and finally, the processing of an EBR-II cold trap.

The completion of the MEDEC Test Program defined the end of the "Operational Experience" period for the purposes of this report. This covered the interval from mid-July 1982 through mid-August 1983.

### SMALL-SCALE SODIUM TESTS

The written procedures<sup>(3)</sup> and the computer-control software that actually directs system operations were intensively reviewed before the initial sodium test. The procedures and related software all required changes to ensure direct correspondence between the procedures' words and the programming. The procedures and software covered such routine processing steps as testing of the lid seal for the M/E vessel, purging and heatup of the M/E vessel, and melting and evaporation operations. (The procedures and software did not yet include validated programming for such priority items as automatic alarms and protective-system responses; the programming for those was completed later.)

Activation of the SPD facility via the test evaporation at 900°F of a small beaker containing several hundred grams of sodium (Fig. 3) took place on July 29, 1982. With the exception of unanticipated sodium deposits on the upper inside wall of the M/E vessel, operations were generally satisfactory. These sodium deposits apparently came from sodium vapor condensation on cool spots in the M/E vessel wall.

A posttest evaluation was held. Conclusions reached were:

- More insulation, and perhaps supplemental heaters, were needed on the upper portions of the M/E vessel exterior, particularly in the lid-clamp area. Additional heating had to be installed carefully and judiciously, as it must not jeopardize the temperature sensitive elastomeric vacuum-seal O-rings that were considered vulnerable to excessive heat.
- The sodium residues on the vessel wall were removed. These were to be quantitatively analyzed (by weight) for evaluating the distribution of sodium deposits.



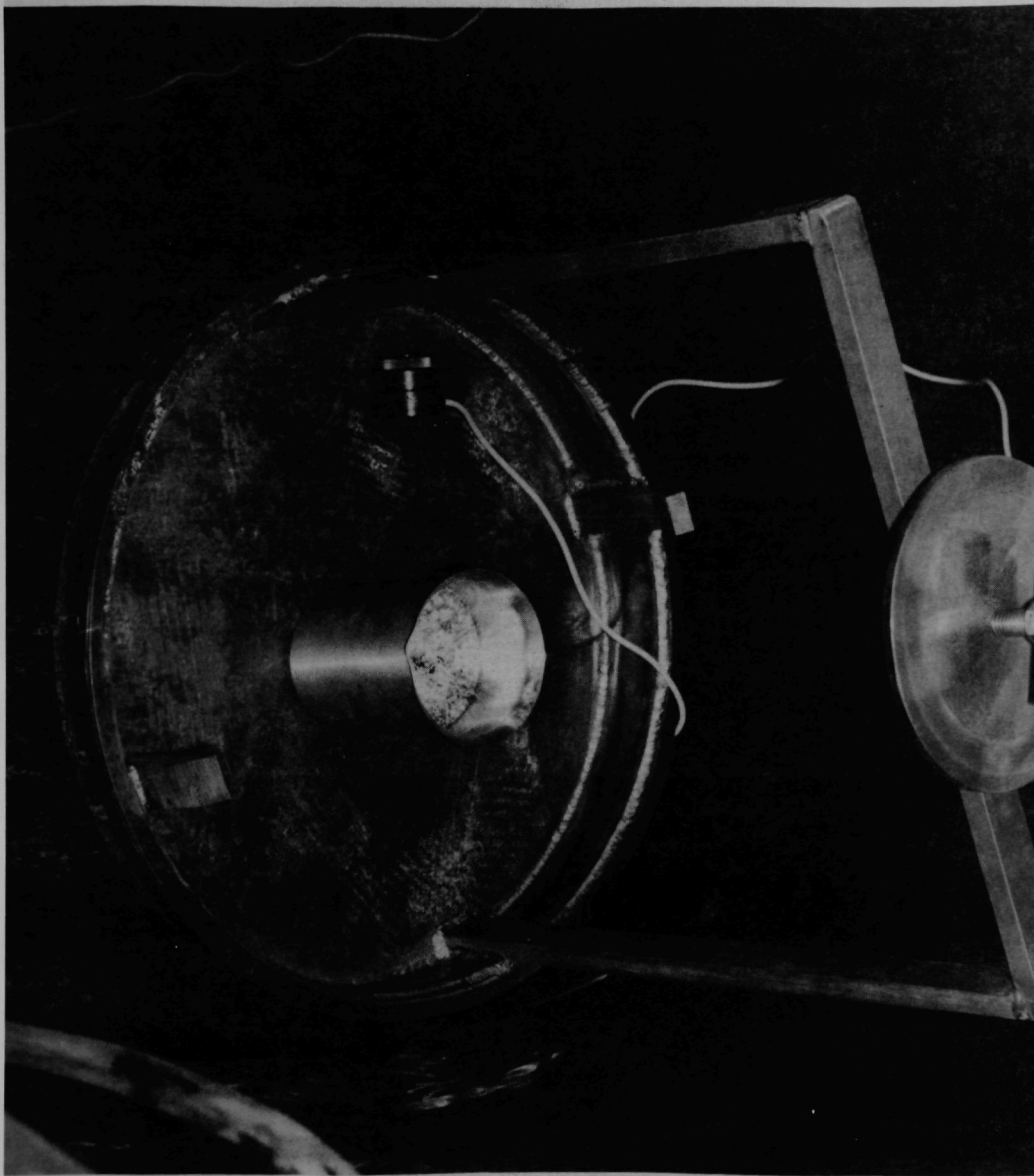


FIG 3 oed  
SMALL-SCALE SODIUM TEST SAMPLE





- Special test thermocouples were to be installed on the inside and outside of the M/E vessel near the lid-clamp area to evaluate local temperature distributions. (Installation of special test thermocouples, when needed, proved to be an invaluable aid throughout the MEDEC Test program.) After nonsodium heatup tests and thermal insulation modifications, etc, the initial sodium test was to be repeated.

After more thermal insulation was installed on the M/E vessel, the small-scale evaporation test was repeated. Some sodium deposits again were evident in the upper interior part of the M/E vessel and on the underside of the M/E vessel lid, but slightly less so than in the initial test. Apparently very little of the vaporized sodium was passing out of the M/E vessel system into the condenser.

These two test results commenced a repetitive cycle of small-scale evaporation tests of a sodium sample contained in a beaker. These were primarily directed to getting evaporation rate data. Another goal of these tests were also to minimize or eliminate sodium vapor condensation in the M/E vessel and to encourage the proper migration of, and condensation of, sodium vapor in the condenser. This latter goal was eventually achieved in piecemeal fashion over a period of about 4 months by adding thermal insulation and supplementary heaters, reprogramming M/E vessel heater energization/ deenergization, changing processing techniques and procedures, and making plant modifications.

Finally, the unwanted sodium condensation in the M/E vessel was eliminated. An additional benefit of this was that the new thermal insulation minimized heat-flow losses from lower-down portions of the M/E vessel. These heat flows were now prevented from affecting the elastomeric O-ring seals in the M/E vessel's lid. This decreased the latter's vulnerability to heat damage which had been of much concern during facility design. Subsequent tests proved that below a temperature of 800°F in the M/E vessel, a loss of air-cooling flow to these lid seals would not result in seal failure.

Also during this small-scale testing period, the sodium vapor-flow baffles in the condenser were redesigned by the SPD Operations staff and rebuilt to enhance vapor condensation. More baffles were added, and the



clearance between baffle and the cooled condenser-wall ID was drastically reduced. The baffles were also redesigned to eliminate the previously installed slots (to accommodate the thermowells) which bypassed sodium vapor. New baffles were spaced so that by careful preplanned movements during the installation of the baffle, interference with the protruding thermowells was eliminated.

In parallel with this small-scale testing efforts, other ongoing activities were completed. These included:

- Installation of floor grating over the entire process equipment pit area. This gave operators walking access to the process equipment and eliminated the necessity to climb about on hot process equipment.

- A study to evaluate a new apparatus designed to determine the endpoint of M/E evaporation was commenced. HFEF design engineers conducted this evaluation in the ANL-W Laboratory and Office Building vacuum laboratory. It was (and still is) a low priority, and ongoing study.

- Commenced construction of building extensions to the SPD facility (Building 789) to support eventual conversion to a low-level radioactive sodium-waste facility.

- Reperformed the process-equipment pit fire suppression test<sup>(8)</sup> using argon gas as the fire-suppression medium. The test was successful even though manual deenergization of certain components, such as process cell blowers, was required. (Automatic control over these atmosphere-moving components in the event of the need for gaseous fire suppression was eventually effected by the process computer.)

- Modified the installed condenser heaters and modified the heater system to melt the sodium that collects in the bottom of the condenser. Also installed additional heaters on the condenser drain tank.

- Installed a demister section in the top interior of the condenser. This keeps sodium vapor from migrating into the suction line to the high-vacuum pump.

- Completed a few acceptance tests that had not been entirely completed prior to HFEF takeover of the facility, including some acceptance work on the calciner.



- Continued development of computer software to achieve automatic control of routine process operations.
- Redesigned the lid clamps for the M/E vessel lid. The originally installed clamps were becoming exceedingly difficult to operate and, in at least one case (experienced later on during "crevice" testing), the tightened clamps exerted insufficient clamping pressure. This caused inleakage to the M/E system under vacuum and aborted one test run.
- Made a basic change in evaporation mode operations by applying vacuum to the M/E system at temperatures above 400°F during heatup, evaporation, and cooldown. This change significantly reduced the amount of sodium condensed in the top flange area of the M/E vessel and virtually eliminated sodium vapor blow-by in the condenser.

#### Multisample Testing

After the problem of sodium condensation in the upper part of the M/E vessel had been solved, multisample evaporation tests using four or five beakers of sodium simultaneously were performed. These tests evaluated the evaporation rates at various regions inside the vessel. After several of these tests were performed, the evaporation rates measured at different parts of the M/E vessel corroborated the heat distribution data taken during the latter portion of acceptance testing. This evaporation testing also confirmed the need for even more heaters to be added to the bottom of the M/E vessel.

Piping distortions were noted during hot-plant conditions when piping positions were compared with cold-plant locations. These deflections were analyzed,<sup>(9)</sup> and corrective action was taken. Only in one instance was there a remote possibility of piping overstress if no corrective action had been taken.

The concerns for the inadequacy of the originally installed electrical heat tracing to heat up the sodium piping to proper temperatures were renewed. A representative section of the sodium piping was set aside as an electrical heat-tracing test-bed to thoroughly evaluate whether the apparent inadequacies were in the heat tracing, or in the indicating/controlling thermocouples. It turned out to be in both.



## MEDEC TESTING ON SODIUM-FILLED CREVICES

An important aspect of the MEDEC test program was to ascertain how effective the SPD process system was in removing sodium from narrow and deep crevices in test specimens. Previous studies<sup>(10)</sup> indicated the MEDEC process was probably the only method (of several alternative methods) to completely remove sodium from narrow crevices.

As recounted hereafter, the SPD MEDEC process removed sodium from crevices 8 in. deep and from a gap that was 2 in. long and only 0.005 in. wide at the narrowest dimension. M/E processing also removed nearly all the sodium film from a crevice of comparable depth-length dimensions whose gap-width was comprised essentially of metal-to-metal surfaces. This crevice testing program was also eventually extended to cover evaporation tests from sodium-filled mesh, and evaporation through sodium-reservoirs with "chimneys" as discussed later on under "Sodium Vapor Conductance Testing."

Each crevice test sample specimen was a bolted-together array of rectangular metal plates (3-1/4 x 8-11/16 in.) with changeable spacer inserts. Several test arrays, dismantled to evaluate the effectiveness of the crevice filling methods, are shown in Fig. 4. The inserts were made of removable shim stock of varying thickness and varying configurations. When bolted together into individual assemblies, the 15-unit array of test sample assemblies could provide the following rectangular crevices:

- 2 x 8-11/16 in. (open on both ends)
- 2 x 8 in. (closed on on one end)
- 2 x 6 in. (closed on on one end)
- 2 x 4 in. (closed on on one end)
- 2 x 2 in. (closed on on one end).

Spacers providing the gap width were 0.105 in. (12 ga shim stock), 0.060 (16 ga), 0.036 (20 ga), 0.023 (24 ga), 0.010 in., 0.005 in., and 0.001 in.

A spacer shim was left out and plates were bolted together and dunked in the sodium bath for a weight "blank" in each test run. This gave the metal-to-metal-surface specimen configuration, mentioned earlier.





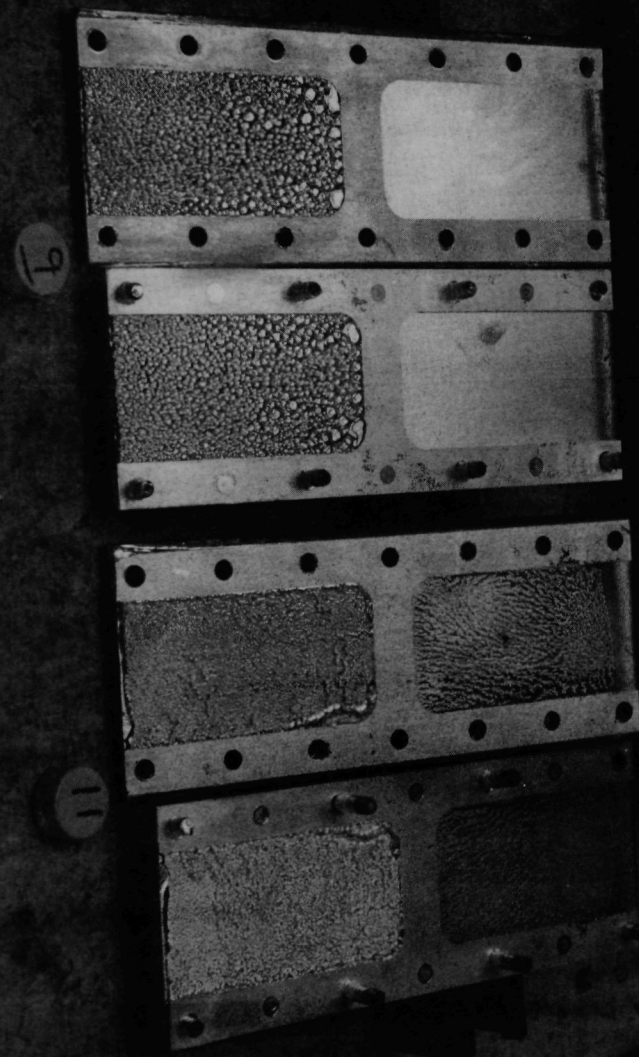


FIG 4 oed



The crevice-test assemblies were filled with sodium in a sodium-bath apparatus located in an inert-gas glove box in the analytical laboratories at ANL-W. Some specimens (shown in Fig. 4) were taken apart to confirm the crevices were entirely filled. After M/E processing, the test assemblies were returned to the analytical laboratories for posttest disassembly, inspection, photographing, and weighing.

Three test runs were made using crevice test assemblies, with specimens with the gap width of 0.105 in. processed first. The test was repeated with a gap width of 0.036 in. and finally 0.005 in. In each test run, some specimen-plate assemblies were oriented vertically (gap up), some horizontally (flat side of plate up), and some at 45° from vertical.

At the completion of the 0.005-in. gap-width test, only one more-stringent crevice test with a 0.001 gap-width could have been performed. But since all sodium specimens had consistently been free of sodium after processing, including mating bolt and nut threads which had been observed to be wetted with sodium, it was decided to move on to other testing. The surface-to-surface weight "blank" mentioned earlier had been performed during the last two gap-width tests.

In parallel with this ongoing crevice testing, there was, in SDP Operations, a great deal of other parallel activity going on. These other-than-crevice-testing activities were focused on the need to refine processing techniques and equipment to support the upcoming bulk sodium and calcination tests. They included the following:

- Continued testing on the sodium-evaporation end-point apparatus.
- Continued software development for the melt/evaporation process control programs, and for the upcoming calcining bulk-sodium and sodium-transfer operations.
- Continued thermocouple/electrical heat tracing studies on process piping. These confirmed that most of the electrical heat tracing on lines and valves was insufficient for the upcoming sodium service. There was a 100° error depression (in the 400°-500° piping temperature range) between actual pipe temperatures and those indicated by specially installed test thermocouples. On the basis of this testing, installation began on new heat tracing and new control thermocouples as follows:



- Continued other minor modifications for plant improvements.
- Commenced conceptual design for a new level-measurement apparatus for the sodium storage tank.

- Performed a test to get data on the evaporation rate from a sodium pool in the interior bottom of the M/E vessel.

- Installed a backfit modification to all the M/E vessel lid clamps. Significant numbers of these clamps had begun to fail in service.

In one case, insufficient clamping pressure caused some inleakage while the M/E vessel was under vacuum. The test sequence was stopped. Posttest inspection showed the crevice evaporation was complete even though the required evaporation time, as calculated, has not been reached.

- During an investigation of the inleakage problem, some warping of the M/E vessel seal-flanges was observed during elevated plant temperatures. This phenomenon, along with the difficulties experienced in changing the elastomeric O-rings in the metal seal ring led to a recommended design change. This modification is expected to include a redesigned seal assembly with slightly larger diameter O-rings and a different O-ring groove configuration. Permanent thermocouples that would measure seal ring temperatures should also be installed.

- Began a study of electrical problems in the M/E vessel heater network. (11)

- Added an additional 3 kW of heaters to the bottom dish of the M/E vessel, bringing a heater capacity to 6 kW to the vessel's bottom head.

- Changed cooling-air ducting to the condenser cooling shroud to provide counter-flow cooling with respect to sodium vapor flow.

## CALCINATION OPERATIONS

As inferred in the previous sections, throughout most of the MEDEC Test program there were two parallel paths of facility operations and activities, with one path leading directly to the immediate test objective. The other path was really a network of activities; most of them involved preparing for the test objectives following the immediate one, plus a



few continuous/repetitive activities such as the sodium-evaporation apparatus endpoint study, procedure and software development, and ongoing repair and upgrade of plant equipment.

This multipath SPD operations approach probably reached the peak of intensity during preparations for calcining.

Not only was the procedure and software development for calcining and sodium transfers especially complex, but the electrical heat tracing and control thermocouple installations on all the sodium piping had to be refurbished and checked out for proper operation. In addition, two new pieces of equipment, the sodium storage tank and the calciner plus the interconnecting piping and instrumentation were being put into actual process service for the first time. This had to be done along with conducting significant repairs on portions of the M/E system which were not being used regularly for the first time in nearly 9 months.

#### Preparation for Bulk-Sodium Operations

The work leading directly to the initial calcination first included bringing the sodium storage tank to a state of operational readiness. To do this involved the following:

- Opening the tank, inspecting it, and removing some miscellaneous debris apparently left over from construction activities.
- Conducting a study to optimize the location of the electrical conductivity probes for determining sodium level at various locations in the tank, and modifying the probes to better suit operational needs. Also, provisions were made for later installing an eddy-current level detector for backup determination of sodium level in the tank. This would permit continuous sodium-level monitoring and would back up the discontinuous level probe system.
- Adding more thermal insulation to unlagged portions of the sodium storage tank.
- Splitting the electrical heater control system in the sodium storage tank to control tank heating via its bottom heaters. Heretofore only the top of the tank was sufficiently heated in process operations, and the tank's bottom temperature didn't even heat up to the sodium melt-point temperature.





- Completing the necessary software to support certain computer-controlled or manual process operations; specifically for transferring sodium between the M/E vessel, the sodium drain tank, the storage tank, and sodium transfers to the calciner.

- Installing a separate nitrogen pressurization line to the sodium drain tank. The tank design had provided a piping stub for this service. SPD operators also modified the sodium-level probes in the sodium drain tank to better suit operational needs, and added additional heaters and thermal insulation to the exterior of the drain tank.

### Bulk Sodium Operations

After completing the foregoing work, the MEDEC process system was ready for the introduction of bulk sodium into the SPD plant. A 55-gal drum of sodium was obtained from the EBR-II sodium storage, the drum loaded into the M/E vessel, and subjected to M/E processing. Following this, a series of liquid sodium transfers were made between the M/E vessel, the drain tank, and the storage tank. These transfers were made to calibrate sodium-flow instrumentation. Similar (but simulated "no-sodium") transfers were made to the calciner.

### Calciner Preparations

To prepare the calciner for upcoming operations also required much preparation, including:

- Repeating the original acceptance-test leak testing on the calciner drum to higher performance criteria, and repairing the leaks found.

- Installing electrical insulating standoffs on calciner heater terminals to effect better electrical heater circuit insulation.

- Installing a bypass valve in the nitrogen supply line to support a calciner shut-down dry-nitrogen purge flow, and initiating such gas purging service.

- Performing extensive "no-sodium" heatups of the calciner, the sodium storage tank, and the sodium piping to check procedures and software, and equipment modifications.



- Developing a procedure and fixtures for charging the empty calciner drum with a bed of sodium monoxide granules. This bed would act as a "seed-bed" substrate upon which to "grow" the new monoxide granules from sodium additions.

- Permitting better viewing of the monoxide granules in the drum by installing a clear Lexan site glass in the calciner drum. This replaced the original somewhat-translucent plate-glass viewing port.

- Calculating process variables ranges, or setpoints. Some of these required extrapolating ANL-E calciner test data.

As a result of the foregoing, the calciner injection line was removed and the calciner drum was opened. The drum's interior was cleaned of miscellaneous debris and 300 lb of commercially manufactured sodium monoxide manually added (Fig. 5) to the drum. Rotation and drum unloading tests were then made to calibrate and check out equipment.

#### Initial Calciner Operations

Following reinstallation of the sodium injection line, a single small batch of sodium (~ 1 gal) was injected into the calciner. Through the site glass, confirmation was seen that the metallic sodium was well dispersed among the monoxide granules, and oxidation of the sodium proceeded as planned during the subsequent calcining phase.

This calcination of sodium fulfilled one of the original test objectives of the MEDEC Test program, and met an ANL commitment to calcine sodium in the SPD facility by March 31, 1983.

#### Postcalcining Rework

At the completion of the initial calcination, the calcining and support systems were inspected. A number of deficiencies were found, some of which required extensive correction. This was not surprising. The preparation work for calcining was the first time much of the system had been extensively operated since initial acceptance testing. The deficiencies found included:

- Misalignment of the trunnion drive shafts.





FIG 5 oed  
LOADING SODIUM MONOXIDE INTO  
CALCINER DRUM



- Deviation from true-cylindrical surfaces in the drive roll rings, resulting in excessive wear in the rings and erratic drum rotation rates.

- Loose pillow-block bearings in the drum's drive test and no provision for adjustment or maintaining alignment in these pillow blocks.

- Excessive axial play in the drum during rotation.

- Extreme wear in the Muntz metal seals between the rotating drum ends and the adjacent stationary calciner structure.

- The calciner RPM indicator did not supply proper feedback.

The first three deficiencies required the efforts of ANL-W machine shop repairmen to correct. The seals, however, were initially a problem during acceptance testing and continued to be a problem throughout the MEDEC test program.

Not only was seal-wear excessive, but minute and annoying amounts of sodium monoxide particulates escaped through the seals, and pervaded the cell atmosphere in the vicinity of the calciner during calcining.

These seals must be redesigned prior to radioactive low-level sodium waste calcining!

To return to the subject of this section, in the postcalcining rework the calciner seals were removed. They had the lubrication grooves deepened and more lubrication fittings installed for better lubrication distribution. They were relubricated (after reinstallation) with a new chemical resistant grease. The seals seemed (upon leak-testing) to perform better.

- During the seal repair work, it was noted that the sodium injection line inside the calciner actually sloped upward. This was contrary to design. This improper slope was due to the weight of the entire sodium-injection line causing deflections in the flexible expansion bellows attached to the related seal assembly. This allowed sagging of the line. To correct this upward-slope problem, the entire line (from its high point outside the calciner) was redesigned and rebuilt to provide adequate slope downward.

In addition to this extensive calciner-refit work, other operations were going on in parallel. These included:





- Removed all thermal insulation from the exterior of the M/E vessel to troubleshoot<sup>(11)</sup> the persistent problems with the installed M/E vessel's exterior electrical resistance heaters. During the latter portions of the small-scale sodium tests, new patterns of sodium vapor depositions indicated some cold spots in the vessel's heating system resulting, presumably, from heater failure. Heater difficulty was corroborated by measuring electrical load imbalances between various heater circuits.

After thermal insulation removal from the vessel, the heaters were all found to be intact and failure-free, but many failures had occurred in the electrical jumper-wire connections. New high temperature wiring was installed, and all connections were silver-soldered to ensure continuity of service.

- Repaired a number of other heaters in the M/E process system which had failed, or showed signs of doing so.
- Improved the thermal insulation on process equipment where operating experience so indicated.
- Continued testing the sodium-evaporation endpoint-detection apparatus, as well as procedures and software development for M/E operations, transfers of sodium, and calcining.
- Investigated a blockage that had shown up in the drain line from the condenser to the sodium drain tank. After removing insulation and heat tracing and cutting into the line, the blockage (when removed) was found to be a saw-blade segment left over from construction. The system was subsequently rewelded and placed back in service.
- Continued minor plant repairs and upgrade modifications.

#### Full-Scale Calcination Tests

With the calcining repairs complete, activities focused on the means for putting sufficient sodium in the system to build the calciner bed up to the requisite 20% volume percent. This is defined as the sodium monoxide occupying 20% of the total drum volume.

Studies were made of the amount of sodium necessary for the required monoxide volume. Two available 55-gal drums of sodium were then obtained from the EBR-II project storage at ANL-W.



A permanent (but removable for cleaning) grating was designed and installed in the bottom of the M/E vessel near the junction of the cylindrical wall and the dished bottom. This put any sodium-bearing workpiece that is loaded into the vessel at a higher elevation in the vessel than the originally installed workpiece location. The grating thus positions the workpiece away from the poorly-heated dished bottom of the vessel. The workpiece is thus better positioned to receive radiant energy from the well-heated cylindrical sides of the vessel.

The two drums of sodium were loaded into the M/E vessel and M/E processed. During the evaporation/condensation operations, it was noted that certain condenser thermocouple indications (or lack of them) might be indicative of the evaporation endpoint, but this appears to be valid only for bulk sodium operations. (This conclusion will be investigated in proposed post-SPD test-program operations (q.v.) or production testing (q.v.) discussed subsequently in "Future Operations.") The resultant liquid sodium was then transferred to the sodium storage tank.

These bulk sodium operations also underscored the need for better heating for the underside of the dished bottom of the M/E vessel. Evaporation of the small pool left in the bottom of the M/E vessel after pressure-transfer of melted sodium to the storage tank seemed to take excessive time.

Prior to the extensive calciner run to bring the monoxide volume up to 20 v/%, a series of small-volume sodium injections/calcinations were performed. These were made to check out instrument calibrations, operating procedures, and software applicability, and to groom the entire calciner process system. The small injections also corroborated process variable setpoints previously calculated or estimated by the SPD Operations staff.

The need for viewing inside the calciner drum during process operations had been pointed out during these small runs. Trying to view the tumbling bed's behavior with a flashlight, through the drum's small single site glass while the drum was rotating, was an impossible task. The calcining gas outlet (off-gas) line was therefore modified. A high-temperature (oven range) incandescent lighting fixture was improvised



and installed. This interior drum lighting proved to be of immeasurable benefit during subsequent large-scale calciner operations even though viewing through the slowly moving sight glass was only possible through about 270° of a drum rotation. For example, the distribution of liquid sodium over the bed as a function of sodium batch size, injection time, and sodium-tank pressurization could be actually seen, and changes to process variables made as required. Also, the thoroughness of injected sodium-bed mixing could be observed. Incipient plugging in the end of the sodium injection line could also be watched for.

As processing continued, the sodium batch size (and corresponding bed mixing and oxidation time) was increased by a predetermined schedule devised by the SPD Operations staff.

#### Postcalcining Evaluation

At the completion of the extended calciner run, the following overall observations were made:

- Product quality was beyond expectations, with free sodium analyzed to be ~ 100 ppm and sodium peroxide  $\text{Na}_2\text{O}_2$  ~ 0.6%. This product quality (for waste-disposal purposes) is better than that of commercially manufactured monoxide, or that of the monoxide product derived from laboratory scale calcination studies by ANL-E.
- There was good correspondence between calcination endpoint as determined by the various process instruments.
- As mentioned, the improvised internal drum lighting system was very helpful in helping operators to evaluate ongoing calcination work.
- Product carryover out the effluent gas line and deposited in the knockout drum/cyclone separator was not excessive.
- The sodium storage tank modifications and the revised electrical heat tracing on sodium process lines and related temperature performed perfectly, as did the related computer software.
- Calculations of the expected bed volume (based on bulk granule density estimates) were confirmed.

Some negative aspects observed in the extended calciner run (which did not detract measurably from the overall success of the operation) were:



- At ~ 20% bed volume, the large sodium batch injected sodium-wetted the entire bed. The bed then acted as a cohesive mass rather than an uncohesive angle-of-repose-bed of tumbling granules. The friction drum-drive had to lift nearly the entire bed mass intermittently until it eventually fell back to the bottom of the drum. As mixing continued, the cohesion diminished somewhat but was still very noticeable. This unexpected cohesive bed behavior overloaded the drive machinery. This situation will have to be dealt with in some manner.

- The RPM tachometer feedback requires additional work to make it function properly. (It has yet to perform as designed).

- The drum seals performed well for awhile, but again - eventually - leaked particulates into the process cell atmosphere. This occurred more during the latter part of full-scale calciner operations. More frequent lubrication of the seals is expected to improve their performance and prevent/minimize monoxide introduction into the cell's atmosphere.

- The need for continuous backup sodium level indication in the sodium storage tank (via the proposed eddy current apparatus) was confirmed.

- Some whitish- and blackish-colored lumps were observed in the monoxide bed, but were not prevalent in the bed. These lumps apparently result from buildup of sodium-wetted monoxide on interior drum surfaces. These buildups break off eventually and are somewhat size-reduced during subsequent drum rotation. Perhaps a redesign of drum internals, or improving the surface finish of internal surfaces to prevent such buildup, or use of a stainless steel-lined drum could prevent such lumps. Redesign of the internal mixing flights to improve mixing should also be considered.

The finely divided product carryover in the calciner gas effluent line was not effectively removed by the knockout drum and cyclone separator. The carryover deposits eventually plugged the O<sub>2</sub> analyzer supply line and flow rotameter. The 3-in. off-gas line also plugged on the air side of the pressure control valve. The O<sub>2</sub> analyzer supply line and rotameter were cleaned and a small filter with a replaceable filter element was installed. This apparently corrected the problem. The off-gas line to the vent stack was dismantled and unplugged but the





plugging problem is unresolved and will undoubtedly cause more trouble with future calcining operations. The cyclone separator may be too large to effectively remove the particles with the comparatively low off-gas flow rate. Some in-line dust filtration system may also be required.

Again, in parallel with the ongoing calcining test work, the following activities were performed:

- Preparations, including test procedures software and test fixtures, were being made for processing an EBR-II cold trap (q.v.) and also processing EBR-II blanket rods (q.v.). Both of these items were original test objectives of the MEDEC Test program. Preparations were also made for performing upcoming vapor conductance tests (q.v.).

#### SODIUM VAPOR CONDUCTANCE TESTING

The ability of the MEDEC system to evaporate sodium from surfaces directly exposed to the high-vacuum/high temperature environment in the M/E vessel and, in like manner, to successfully evaporate sodium from sodium-filled crevices, was considered by now to be proven.

With the M/E vessel heater system restored to good order,<sup>(11)</sup> and the calcination test work complete,<sup>(13)(14)</sup> attention now turned to tests of sodium evaporation where the sodium was not directly exposed to the high-vacuum/high-temperature in-vessel environment. Evaporation tests of partially enclosed apparatus was thought to provide insights as to how well the MEDEC system could evaporate sodium from complex equipment containing internal sodium that had complex internal geometries. In such circumstances, any sodium vapor would have to migrate significant distances from the internal pockets of sodium, then conduct itself through piping, tubing or mesh (or other conduits) to the environment of the M/E vessel's interior. Such so-called "vapor conductance tests" were planned for, early-on. They proved to be an important factor in providing practical experience to successfully process full-sized sodium-containing equipment with complicated internal structures, such as the EBR-II cold trap.



To mock up evaporation from a portion of an EBR-II cold trap, a special vapor conductance test fixture was fabricated. A large ( $\sim 2$  liter) stainless steel beaker was fitted with a cover. A penetration in the cover was connected to an open-ended 2-in.-dia pipe "chimney" comparable in length ( $\sim 4$  ft) to the height of the cold trap. The beaker had stainless steel mesh installed in it (like the trap), and 30 in. of the chimney was also filled with mesh. The assembled fixture was partially filled with sodium.

This apparatus was installed in the vessel. It was M/E processed with the "chimney" pointing upwards, along with a sodium-filled "flow simulator" from the ZPPR (Zero Power Plutonium Reactor) Project.

After M/E processing, the "flow simulator" was found to be free of sodium. The evaporation rate from the conductance apparatus, however, was found to be very low. As a result of this testing, more data was considered necessary for the evaluating of the conductance of sodium vapor through metallic conduits when exposed to the M/E vessel vacuum/temperature conditions.

A duplicate of the first mesh-filled conductance apparatus was fabricated and installed in the M/E vessel with the chimney pointed down. This better simulated the cold trap when the latter would be processed in an inverted attitude. In this orientation, sodium draining would also augment the evaporation in removing sodium. The two mesh-filled units are shown in Fig. 6.

In addition, four other smaller beakers were equipped with chimneys of various lengths, diameters, and configurations but did not contain any mesh. The "longest" chimney was an  $\sim 5$ -ft length of 1/2-in. diameter tubing bent in a loop. These smaller vapor conductance test beakers were sodium-filled and loaded into the vessel (Fig. 6).

An evaporation time was chosen for M/E processing to maximize the likelihood of getting pertinent vapor-conductance data.

This second vapor conductance test was performed along with M/E processing the EBR-II blanket rods. The stand for holding the blanket rods in a vertical attitude is shown in Fig. 6.



FIG 6 oed  
VAPOR CONDULTANCE TEST FIXTURES  
(AND BLANKET ROD TEST FIXTURE)





The second conductance test was successful in that all sodium was removed from the inverted mesh-filled apparatus. This raised expectations about the success of processing the cold trap.

Also, the conductance data from the other beakers processed was found useful. Only the mesh-filled beaker with the "chimney" up, and the beaker equipped with the tubing loop still contained some sodium.

This was consistent with the phenomenon that the mean free-path of sodium vapor in the M/E environment is about 1 in. Hence the vapor would have more difficulty in conducting itself through mesh or through tubing with such a small diameter.

On the basis of this second vapor conductance test, firm plans for processing the EBR-II cold trap were made.

In addition to the extensive ongoing preparations for ultimately processing the cold trap (also performed in parallel with ongoing test operations), were:

- Installation of in-line filters in calciner-process and instrumentation lines to prevent plugging by monoxide particulates.
- Continuation of final procedure and software development and updates of operational and test procedures, continuing tests evaluating the sodium evaporation endpoint apparatus, and implementing minor improvements to upgrade the SPD plant.
- Receipt, installation, and preliminary testing on the eddy-current apparatus for monitoring the level in the sodium-storage tank.

#### M/E PROCESSING OF EBR-II BLANKET RODS

The second scheduled test objective of the MEDEC Test program was to M/E process sodium-bonded, depleted-uranium EBR-II blanket rods.

Each rod has five 11-in.-long depleted uranium slugs sodium bonded to steel cladding. During reactor operation, heat transfer from the uranium slugs to the cladding is enhanced by this thin annular sodium-bond. It is the removal of this thin, annular, sodium bond that was an objective of the M/E process.





Ten unirradiated blanket rods were obtained from the EBR-II Project, and cut in the ANL-W Analytical Laboratory labs at various prescribed locations according to the directions of the SPD Operations staff. Some rods had only one end cut off, and some had both ends cut off. Some rods had the cladding cut in the center and some had the cladding cuts at the junction of adjacent slugs. Placed in fixtures, the rods or rod segments were processed along with the vapor conductance apparatus as discussed in the previous section. The rod holder fixture, without rods or rod segments, is shown in Fig. 6.

All blanket-rod test configurations with the opened-end of the blanket rods cladding pointed downward were found after processing to be free of sodium, but other configurations were not. The configurations that were free of sodium resulted in easy separations of the slugs from the related cladding segments. There was no uranium contamination of the M/E process vessel, nor of the conductance apparatus also coprocessed with the blanket rods.

The second test objective of the MEDEC test program was therefore met.

It appears that the most effective mode for any future processing of such blanket rods would be to cut off the bottom end of the blanket rods and process the rods in a vertical orientation.

#### COLD TRAP PROCESSING

The capstone of the MEDEC Test program was the processing of an EBR-II cold trap. Not only was this a demonstration of the effectiveness of the M/E system to remove sodium from an item with complicated internal structure, but it also proved the M/E system's ability to remove sodium from a full-sized LMFBR component.

Among the several EBR-II cold traps available, the one chosen for processing was a cold trap that had been used in precritical operations in the EBR-II primary system. As such, it contained a full complement of sodium hydride collected on the internal stainless steel mesh but no tritium. The cold trap was a cylindrical vessel of stainless steel



~ 43-in. high x 32-in. dia with flat ends. (All other EBR-II cold traps have dished ends.) Wrapped around the periphery of the cylindrical section was a stainless steel cooling jacket, with some internal cooling piping in the jacket. The horseshoe-shaped economizer had been removed from the top of the cold trap and the sodium inlet and outlet lines, and a sodium fill line had been cap welded.

The cold trap had been stored in the ANL-W desert laydown area with other sodium-containing equipment for about 15 years.

The cold trap was retrieved from desert storage. The exterior was steam cleaned, and miscellaneous material (such as old junction boxes) removed.

No records existed to confirm whether or not NaK (the potassium/sodium eutectic alloy) remained in the cooling jacket or the jacket's integral cooling coil.

By means of several tests, NaK was found to be in both. Serious consideration was given<sup>(12)</sup>, to coprocessing the NaK (along with the cold trap's sodium) through the MEDEC system. It was decided instead to remove all the NaK prior to M/E processing. Via special procedures, approximately 35 gal of NaK was pressurized-gas transferred from the cooling jacket and associated cooling coil and burned.

The cold trap was to be processed in an inverted position. This would permit melted sodium to drain out of the meshed portion of the trap. Liquid sodium and vapor would exit the trap from the uncapped 2-in. dia inlet/outlet lines and 1-in. dia fill line. An angle-iron structure was therefore designed and welded to the top of the trap to serve as a processing setdown stand.

With the trap cleaned, its NaK removed, and the setdown stand installed (Fig. 7), the trap was weighed and then installed into the M/E vessel on June 28, 1983. The lines were then uncapped to support sodium removal.

The actual M/E processing commenced in early July and required about 10 days of round-the-clock process operations. After plant cooldown, the trap's internals were given a preliminary examination while the trap was still submerged under argon gas in the M/E vessel. No free sodium could be seen through the piping apertures.







The trap was set down outside the vessel in its normal orientation. More visual inspections through these piping apertures, plus thrusting sharpened rods through the meshed volumes to probe for sodium residuals, indicated all sodium masses had been removed.

The top of the trap was cut open, and the top of the trap was lifted off. Sodium oxide residues made this separation somewhat difficult. This top-removal separation included the meshed portions of the trap along with the cylindrical baffle between the outer annular meshed portion (on the sodium "inlet" side) and the inner cylindrical meshed section. No bare sodium could be seen at any time, but the meshed sections seemed to be well-filled with sodium oxide, and perhaps other sodium materials.

Photographs were taken, and samples taken of the oxide, from all meshed portions. The free sodium in most of these oxide samples analyzed at less than  $\sim 0.1\%$  free-sodium.

In parallel with this cold trap test work, the following other work was being done:

- Installed and checked out the eddy-current apparatus for level measurement in the sodium storage tank.
- Completed all the extensive software (some 75 different programs) for the computer-controlled MEDEC processes, and the related software documentation and editing.
- Implemented the EPROM module (Erasable Programmable Read Only Memory) to direct the process control system.
- Continued compilation of weekly and monthly status reports on facility operations, and of preliminary test reports.

The meshed sections and the cold trap vessel parts were finally water washed to remove the sodium oxide/hydroxides before the items were disposed of as scrap. Some popping noises, characteristic of water-sodium reactions, were heard when water-washing some meshed segments, but the apparent residual sodium probably was not more than a few grams.

With the water-washing of the cold trap components completed, the third and final objective of the MEDEC Test program had been achieved.





## IMPROVEMENTS

This section will discuss improvements already achieved and those yet to be achieved. It will not, in general, discuss proposed modifications to be made for converting the facility to low-level sodium waste processing.

Changes to the facility or equipment found necessary during acceptance testing were documented in Ref. 1.

Changes found necessary after the HFEF involvement in acceptance testing were also documented by using "Change Request Forms-Sodium Process Demonstration." Many of these changes were made, for example, to correct wiring errors, and in general, to make equipment work "like it was supposed to." Other changes were made to make-ready the facility for operations. A listing of these "Change Requests for SPD" is included in Table 1-id.

When acceptance testing was complete, and HFEF Operations assumed responsibility for facility activities, any additional modifications were requested using the "Modification Proposal" (MP) system administered under the HFEF Management Plan (12).

Many of these latter proposals, too, were initiated just to get the facility/equipment to meet design expectations, or to provide well-designed test fixtures. Other MP's were initiated with the expectation that facility effectiveness, ease of maintenance, and safety of operations would be enhanced.

A complete listing of these HFEF-initiated modification proposals is contained in Table 2-id. Almost all items listed have been completed, and several items were cancelled.

A vigorous attempt is being made to document all facility changes, however minor, either with MP documentation, or (for minor changes) to red-line corrections to construction or installation drawings.

The account of how improvements were generally conceived and implemented is reported in the "Acceptance Testing" and "Operational Experience" sections. Of the improvements already made, the ones considered of most significance to the SDP are listed below in unranked order.



Table No. 1 (id)

Sodium Process Demonstration Change Requests

<u>Change Request No.</u>	<u>Description</u>
CR-1	Rewire O <sub>2</sub> analyzer and calciner speed sensors
CR-2	Install temporary thermocouples on heatup test fixture in the M/E vessel
CR-3	Provide monoxide-sampling capability in calciner
CR-4	Rewire high-temperature interlocks
CR-5	Rewire analog/digital converters
CR-6	Add isolation valves to O <sub>2</sub> analyzers
CR-7	Design spreader bar for lifting the M/E vessel lid Provide more lifting capacity in the jib hoist
CR-8	Enlarge cooling air holes in M/E lid-seal ring
CR-9	Install charger for emergency-generator startup battery
CR-10	Modify wiring to close vacuum-pump block valve
CR-11	Rewire valve V-402 to different "Fail" mode
CR-12	Vent discharge of seal-chiller unit to outside
CR-13	Eliminate Mill-Lane valve to cryo pump
CR-14	Add more tracing to sodium lines and valves
CR-15	Add more heaters to condenser
CR-16	Add 3 kW of heating to M/E vessel bottom
CR-17	Add heaters to bottom of sodium storage tank
CR-18	Install additional air-conditioner capacity to cool cell during operations
CR-19	Convert fire-suppression system for process cell equipment pit to argon service
CR-20	Modify seal-cooling system to eliminate upward temperature drift



Table No. 1 (id) (contd)

Sodium Process Demonstration Change Requests

<u>Change Request No.</u>	<u>Description</u>
CR-21	Install protective guards on protuding thermocouple wells in M/E vessel.
CR-22	Lower the inlet to the condenser blower exhaust system to near the floor of the pit.
CR-23	Install relief valves in N <sub>2</sub> dewar piping.
CR-24	Thermally insulate and supply building heat to auxiliary Bldg. 789A.



Table 2 (id)

Modification Proposal List for SDPM.P. Number

D-1	Provide test fixture for internal M/E vessel heat/cool-down tests (for PWS 3-11)
D-2	Design/fabricate test specimens for sodium removal
D-3	Design/fabricate M/E vessel load/unload equipment
D-4	Design/fabricate and install sodium evaporation endpoint apparatus
D-5	Modify Bldg. 789 architecturally for processing radioactive low-level sodium waste
D-6	Design/fabricate calciner unloading and sampling system
D-7	Provide manual argon and nitrogen purge system to condenser
D8	Design/fabricate seal plug for sodium vapor line
D-9	Improve knock-out drum/calciner alignment
D-10	Install piping bypass around dryers to SPD air compressors
D-11	Install manual throttle valve in N <sub>2</sub> purge line to calciner
D-12	Provide backup gas for air-cooling to M/E seals (cancelled).
D-13	Cross-connect M/E vessel and calciner O <sub>2</sub> analyzers
D-14	Install indicating lights on solenoid valves (SV-212 and SV-213)
D-15	Install cell-door interlock on panel alarm module
D-16	Install check valves in nitrogen supply systems
D-17	Provide means to check operability of cell oxygen meter
D-18	Install pressure regulator in argon fire-suppression system
D-19	Install "Alarm Test" capability on fire-suppression system
D-20	Install floor grating over entire process equipment area
D-21	Connect the computer-originated warnings and alarms to the facility alarm panels
D-22	Provide heater control to the bottom heaters of sodium storage tank





Table 2 (id) (contd)

Modification Proposal List for SDPM.P. Number

D-23	Modify seal-cooling blower to improve seal cooling to M/E vessel lid
D-24	Relocate control transformer and fuses for accessible maintenance
D-25	Install permanent thermocouples in M/E vessel seal ring
D-26	Install protective covers over outside-installed air conditioner units
D-27	Design vapor baffle for M/E vessel interior (cancelled)
D-28	Install supports for additional M/E lid thermal insulation
D-29	Install more heaters on condenser bottom
D-30	Modify condenser baffles and install demister section
D-31	Connect development-system computer to control panels
D-32	Design eddy-current detector for sodium tank level
D-33	Install "Kill" circuit in air conditioner power for fire suppression
D-34	Modify probes in sodium drain tank
D-35	Install function control switches on instrumentation cabinet
D-36	Replace calciner off-gas ducts with piping
D-37	Modify louvers to control temperatures in Bldg. 789A
D-38	Redesign lid clamps for M/E vessel
D-39	Cross-connect N <sub>2</sub> purge piping to M/E vessel drain line
D-40	Design and fabricate thimble and coil for eddy-current apparatus
D-41	Design and fabricate test fixtures for multisample testing
D-42	Design enclosure for opening sodium-containing items before M/E vessel loading
D-43	Install roof-ice deflectors over outside personnel doors
D-44	Modify piping for heatup stress or distortions
D-45	Install nitrogen service to condenser drain tank



Table 2 (id) (contd)

Modification Proposal List for SDPM.P. Number

D-46	Install calciner-shroud lifting fixture
D-47	Install small bypass around calciner nitrogen-supply valve
D-48	Split nitrogen purge system to M/E vessel, and to sodium tank and calciner
D-49	Design either better seals or a container boot over calciner drum seals
D-50	Upgrade heater power connection to M/E vessel heaters
D-51	Upgrade drive machinery on calciner drum drive
D-52	Install workpiece support grating in M/E vessel
D-53	Install emergency lighting in process equipment pit
D-54	Install internal lighting in calciner drum
D-55	Replace temporary lighting and outlets with permanent installations
D-57	Fabricate/install internal light in calciner drum/off-gas line
D-58	Provide sodium sampling capability in the sodium storage tank
D-59	Modify calciner drive to prevent roll-ring/trunnion slippage
D-60	Modify off-gas lines and vent to prevent plugging from particulate buildup
D-61	Redesign M/E lid seal



- Improved heating and thermal insulation to the M/E vessel which eliminated sodium-vapor redeposition.
- Installed a workpiece-support grating in the M/E vessel to more effectively apply radiant heat to all workpieces.
- Redesigned the M/E vessel lid clamps for more reliable and convenient installation and removal of the lid.
- Installed temporary special thermocouples in the M/E vessel seal-ring to monitor seal temperatures.
- Installed a special instrumentation thermocouple system. This was used to supplement and back up the installed operational thermocouples, and in troubleshooting and process-system analysis.
- Implemented a coordinated hardware-software procedures approach for all process-system operations and protective response actions.
- Installed floor grating over the entire process pit area to provide safe operator access to all equipment.
- Installed a manual inert-gas purge and vent service to the M/E vessel condenser and installed a backup M/E vessel vacuum indication. The M/E vent service was also isolated from the rest of the vent system.
- Upgraded and electrified the hoist serving the M/E vessel for lid removal and replacement and loading or unloading heavy workpieces.
- Upgraded insulation and heating to the condenser, sodium drain tank, and sodium storage tank.
- Modified the baffling in the condenser to improve sodium vapor transport and condensation, and installed a demister unit in the vacuum-pump inlet.



- Rerouted the condenser cooling to enhance sodium-vapor transport and condensation.

- Redesigned sodium-level probes in the sodium drain tank and the sodium storage tank and designed and installed an eddy-current detector for continuous monitoring of sodium level in the storage tank.

- Added a separate nitrogen line to the sodium drain tank so transfer of its contents would not affect condenser operations.

- Installed new electrical heat tracing to sodium lines and upgraded temperature-control thermocouples.

- Rerouted the sodium inlet line to the calciner to ensure proper line slope for sodium drainage.

- Modified calciner drum seals for improved lubrication and reduced seal leakage.

- Realigned the mechanical drive train to the calciner drum.

- Installed internal lighting to the calciner drum.

Improvements yet felt necessary for MEDEC processing of uncontaminated sodium are:

- To upgrade the calciner seal system to a zero-leakage system.
- To eliminate the excessive wear on the calciner roll ring friction drive and resultant erratic drum RPM from cohesive bed conditions.

Other modifications may be found necessary during the upcoming "Post-MEDEC-Test Operations" and "Production Operations" discussed subsequently under "Future Operations."





## FUTURE OPERATIONS

Other than mentioning in this section that preparations for converting the facility for processing low-radioactivity-level sodium waste are being made on a funding-available and preliminary design basis, nothing additional concerning this matter will be said. The SPD operators are mindful of this ultimately-to-be-fulfilled processing role, however, and keep alert for changes to be made to equipment and procedures considering the low-level plant conversion.

A proposal has been made (16) discussing post-MEDEC-test program operations whose performance is either mandatory before low-level conversion or highly desirable. This document also suggests that the SPD be used as a clean sodium-waste production facility to MEDEC process certain sodium-bearing equipment items now stored at ANL-W. The argument is made that not only would valuable storage space be freed up, but the SPD facility will garner valuable operating experience before the low-level conversion work.

Some 16 equipment items that are considered to be processable in the SPD were identified. In addition, there are ~ 1100 depleted uranium capsules (unirradiated) from which the EBR-II project would like SPD to remove the sodium bonding.

These proposals (16) are currently being considered by ANL-W officials. Although no estimates have been made of the plant time and related costs involved in these proposals, a cursory evaluation would suggest about 9 months would be required to complete this work.



## CONCLUSIONS

The MEDEC process in the SPD facility has proven to be a well-designed and useful tool. It is effective in tests applied to removing sodium from sodium-bearing items and converting the recovered sodium to storable or disposable sodium-monoxide.

Yet to be proven is the ability of the equipment to operate in a sustained production mode rather than a test mode. This is expected to be done in the next 9 months.

The facility will eventually be modified for processing sodium-waste items containing low-level radioactivity. Assuming that modifications can be made successfully to contain the radioactive materials in operations, such as those involved in rotating equipment or loading or unloading the M/E vessel, the SPD is expected to be equally successful in its new role.



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